**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Application of: Thomas Martin Angeliu : Group Art Unit: 1742  
Serial No.: 10/064,510 : Examiner: G.P. Wyszomierski  
Filed: July 23, 2002 : Response to Paper No. 20041123  
For: METHOD FOR MAKING MATERIALS HAVING ARTIFICIALLY  
DISPERSED NANO-SIZE PHASES AND ARTICLES MADE  
THEREWITH

**DECLARATION UNDER 37 C.F.R. § 1.131**

Thomas Martin Angeliu, the sole inventor of the invention disclosed and claimed in the above-identified patent application, serial number 10/064,510, filed July 23, 2002, Attorney Docket RD-28181, declares as follows:

1. I am the sole inventor of the invention disclosed and claimed in the above-identified patent application, hereinafter called "the invention."
2. The invention was completed in the United States by me or under my supervision prior to the 28 December 2001 filing date of U.S. Patent Application serial number 10/028,735, publication number US20030148042A1. At the time prior to 28 December 2001 when the invention was conceived and reduced to practice I was an employee of the assignee of the application and worked in the United States of America.
3. The patent application serial number 10/064,510 was filed in the United States on 23 July 2002, after the 28 December 2001 filing date of U.S. Patent Application serial number 10/028,735, publication number US20030148042A1.
4. Exhibit A, attached hereto and made part hereof, is a photocopy of a patent disclosure letter dated prior to 28 December 2001 and executed by me on a date prior to 28 December 2001.

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to: Commissioner of Patents and Trademarks, Alexandria, VA 22313-1450 on

2/24/05  
(Date of Deposit)

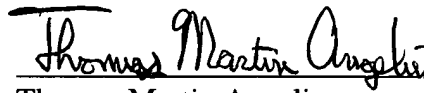
Signature

2/23/05  
(Date of Signature)

5. The invention described therein is descriptive of the invention completed in the United States by me or under my supervision prior to 28 December.
6. The disclosure letter is designated as RD-28181 in accordance with the regular practice of my employer for processing disclosures of discoveries and inventions made by employees in the course of their employment.
7. Exhibit B is photocopy of the Patent Administration Record dated prior to 28 December 2001 recording the opening of a docket for RD-28181. The title and the named inventor correspond to the patent disclosure letter referred to in paragraph 4, above.

All statements made by the declarant herein based on personal knowledge are true and all statements based on information and belief are believed to be true. Furthermore, these statements are made with the knowledge that willful false statements are punishable by fine or imprisonment or both and may jeopardize the validity of the application or any patent issuing thereon.

Inventor and Declarant

  
Thomas Martin Angeliu  
Date: Feb. 23, 2005



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To: Dr. Paul Follansbee, MB213  
Manager, Physical Metallurgy Laboratory  
Schenectady, New York

*Paul Follansbee*  
[REDACTED]

From: Thomas M. Angeliu

**SUBJECT: PATENT DISCLOSURE LETTER**

on: Ultrasonic Processing of Artificial Dispersion Strengthened Materials

Heat resistant steels, nominally Fe-12Cr, are being developed for high temperature applications in power generation applications. One of the challenges for these materials is improving the high temperature properties such as creep rupture strength. In the closing remarks at a conference on heat resistant steels, it was noted that the materials community has reached the end of the road in terms of making slight modifications to Fe-12Cr steels and that a different design philosophy is required.<sup>1</sup> One such philosophy is artificial dispersion strengthening. However, artificial dispersion strengthened materials are exclusively made by mechanical alloying, an expensive process which is not well suited for heavy forgings such as steam rotors. Therefore, novel methods of creating a microstructure of uniformly dispersed, fine and stable particles is required and is the subject of this disclosure.

The method of fabrication in this disclosure utilizes a feedstock material consisting of a very fine powder of nanophase particles.<sup>2</sup> These particles may consist of oxides, carbides, silicides and will have diameters in the range of 10 to 100nm. At the proper time in the melting procedure, the desired amount of nanophase particles will be added to the melt and allowed to uniformly disperse before casting, Figure 1. After casting, the billet is processed with usual procedures.

There is much related work on the dispersion strengthening of metals, however, a majority of this work involves the exclusive use of mechanical alloying to create the fine dispersion required for strengthening. Limited publications on alternate methods to create a dispersion strengthened

material include in-situ oxidation, "Chemical Socking"<sup>3</sup>, oxide powder injection directly into a melt,<sup>4</sup> and the addition of carbides to a melt.<sup>5</sup> To our knowledge, the addition of carbides to the melt is the most closely related to this disclosure, but differs in that the carbides are added as a crushed powder and the particle size is between 1 and 10 microns in diameter. This size is much too large to be effective dispersion strengtheners. By utilizing nanophase feedstock, the dimensions of the dispersed particles will approach the 10 to 100nm range.

To enhance the dispersion of the particle addition, this disclosure advocates the use of an ultrasonic transducer to transmit ultrasonic energy to the molten metal. The energy will be applied through the use of a probe immersed in the molten metal or attached to the mold or surface contacting the molten or solidified metal in some manner, including an ESR ingot or mold bottom. The technique could be applied to vacuum arc melting/ re-melting, electrosag refining, and electron beam melting.

There is much related work on the use of ultrasonic energy in reducing the grain size of castings and increasing the compositional homogeneity of molten metals.<sup>6</sup> However, limited work has been conducted on creating artificially dispersion strengthened materials using ultrasonic irradiation to disperse the strengthening phase<sup>6</sup> and no work to our knowledge on using ultrasonic irradiation with nanosized particles as the strengthening phase.

## REFERENCES

1. Jack Nutting, closing remarks at Advanced Heat Resistant Steels for Power Generation conference, San Sebastain, Spain, April 29<sup>th</sup>, 1998.
2. T.M. Angeliu and C.G. Mukira, "Dispersion Strengthening by Nanosized Particles of Metal Alloys" patent filed in 1999.
3. S. Peng, C. Wu, B. Hu and S. Zhang, "Study on a new oxide dispersion strengthened ferritic steel", Transactions of Metal Heat Treatment, 18, 3 (1997) pg. 101-106.
4. T. Gladman, The Physical Metallurgy of Microalloyed Steels, Institute of Materials, London, UK (1997).
5. P. Bates, M.A. Walsh and S. Price, "Properties of 12%CrMoV High Temperature Turbine Steel Containing FeWTiC", Advanced Heat Resistant Steels for Power Generation, (1998).
6. O.V. Abramov, "Ultrasound in Liquid and Solid Metals", CRC press (1994).

WITNESS AND DATE

READ AND UNDERSTOOD BY:

INVENTOR: Thomas M. Angeliu  
Thomas M. Angeliu  
Physical Metallurgy Laboratory

Date: [REDACTED]

WITNESS: Peter Gundlach

Date: [REDACTED]

WITNESS: Charles M. [REDACTED]

Date: [REDACTED]

**Patent Administration Record**

☒ Docket Opening  
(2 copies--Adm. Clerk)

☐ Filing Application  
(9 copies--Docket Clerk)

☐ Opening and Filing Application  
(9 copies--Docket Clerk)

Docket No. RD-28181

Filed:

Serial No.

**TITLE** ULTRASONIC PROCESSING OF ARTIFICIAL DISPERSION STRENGTHENED MATERIALS

**INVENTOR(s) FULL NAME , OCCUPATION & CRD LAB/BLDG/RM (or list GE operating component)\***

Thomas M. Angeliu, K1-3A51

**DESCRIPTION OF SUBJECT MATTER:**

The method of fabrication in the present invention utilizes a feedstock material consisting of a very fine powder of nanophase particles. These particles may consist of oxides, carbides, silicides and will have diameters in the range of 10 to 100nm. At the proper time in the melting procedure, the desired amount of nanophase particles will be added to the melt and allowed to uniformly disperse before casting. After casting, the billet is processed with usual procedures.

**DATE OF DISCLOSURE LETTER**

**ONE CRD LAB TO BE CREDITED**

**DOCKET REVIEW INFO.**

Physical Metallurgy Lab

Materials Properties Program

**KEYWORDS**

**DESCRIPTION**

**RELATED GE PATENTS AND/OR APPLICATIONS**

**INTERESTED COMPONENT(S)**

GE Power Systems

**PATENT COUNSEL**

R. C. Lampe

**MANAGEMENT CONTACT PERSON**

**DIAL COMM**

**PROJECT NAME**

CRD: S. Balsone

8\*833-4141

GE COMPONENT: P. Schilke

8\*235-7180

**REMARKS** (Indicate Contract No., if any. In use? If so identify Products, facts and dates of Public use, sale or publication.)

**ATTORNEY:** Noreen C. Johnson *ncj*

**DATE:**

**OTHER COPIES TO:** PS Follansbee/SJ Balsone/K. Thorne

**OUTSIDE FIRM  
& OUTSIDE  
COUNSEL:**

\*Give explanation if not currently a GE employee, e.g., Left GE from..., Retiree of..., Consultant for... (list component)

EXHIBIT B